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Technical Report 72

A Single-Crystal Adaptor for the Norelco  
High-Angle Diffractometer

by

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A SINGLE-CRYSTAL ADAPTOR FOR THE NORELCO  
HIGH-ANGLE DIFFRACTOMETER

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Abstract: A description is given of a simple adaptor for the Norelco Geiger-counter diffractometer to measure the X-ray intensities diffracted by a small single crystal.

A simple conversion of the Norelco high-angle diffractometer for use as a single-crystal rotation camera has been devised. A valuable feature of the adaptor is that the standard unit may be converted in a matter of minutes without upsetting the alignment, except possibly for the divergence slit. The adaptor is based upon the principle of completely bathing a small single crystal in a slightly divergent collimated X-ray beam, and slowly rocking the crystal through the Bragg reflection angle, keeping the Geiger counter stationary at the appropriate  $2\theta$  angle. The window of the counter is wide enough to contain completely the diffracted beam which is typically ca.  $0.2 \times 2.0$  mm.

The normal specimen holder is replaced by a long shaft closely fitting the bronze sleeve of the diffractometer, and ends in a screw onto which a Supper goniometer head\* can fit. The other end of the shaft has a graduated circle attached, and is connected with a synchronous motor. The goniometer head must be reduced in length from 6 to 4.5 cm in order to be accommodated with a single crystal, mounted in the usual way on a glass fiber, in the space between the X-ray beam and the diffractometer circle. It is possible to translate the whole

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\* Charles Supper Co., Newton Center, Mass.

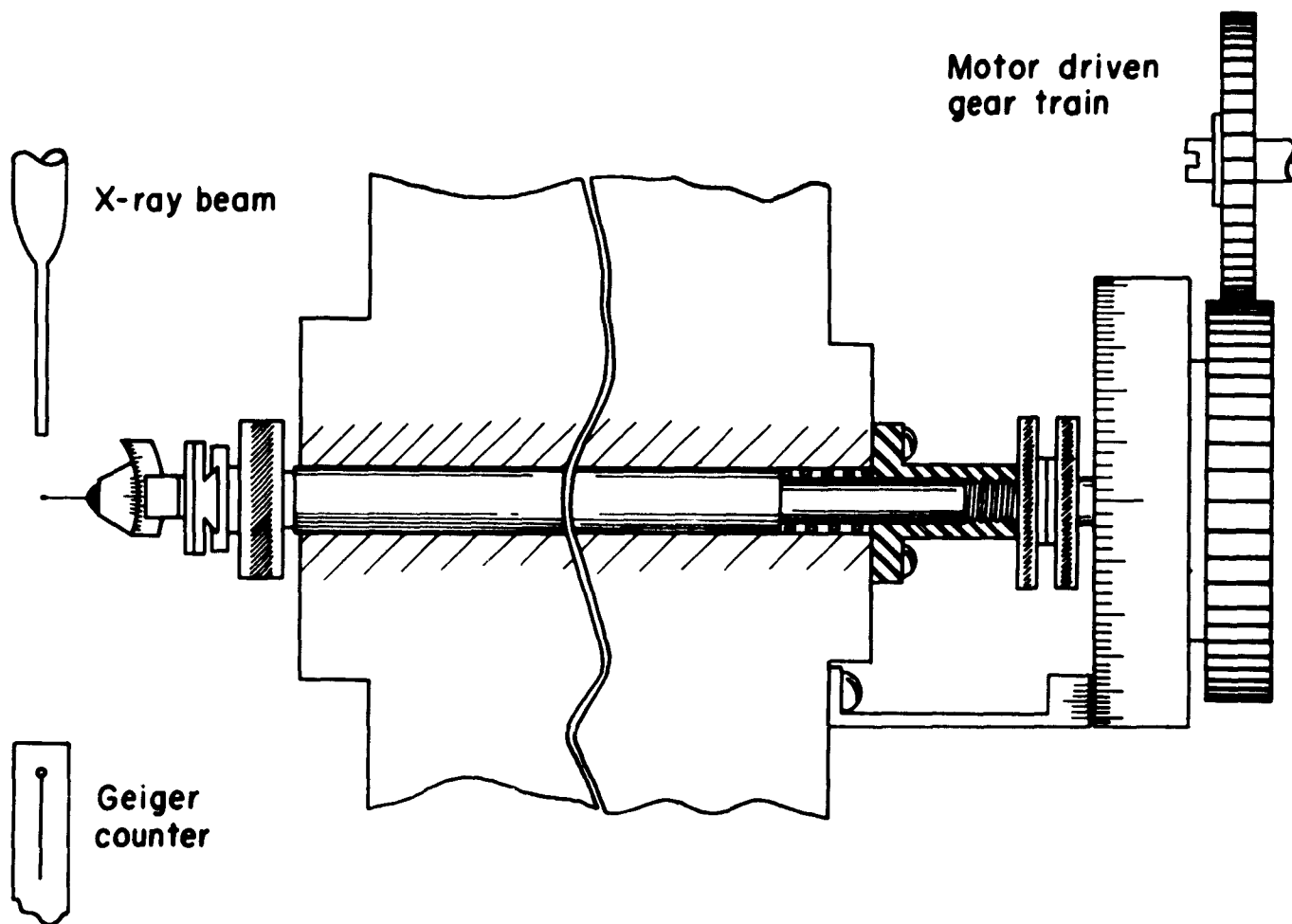


Fig. 1. Diagrammatic view of the single-crystal adaptor for the Norelco High-Angle Diffractometer.

shaft along its length to bring the crystal into the beam. A Bodine motor is used to rotate the crystal with a constant angular velocity of  $1/9^\circ$  per minute. Other speeds are readily obtained by changing the driving gear wheel, which fits directly onto the reduction gear box (Fig. 1). The driven wheel is permanently attached to the graduated circle; direct crystal-setting readings to  $0.1^\circ$  are obtained on it by means of a vernier scale. A Unicam 1mm Weissenberg collimator\* fits directly onto a plate identical in shape with the standard divergence slit plate. A convenient way of centering the collimator is to rotate

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\* Unicam Instruments Ltd., Cambridge, England.



Fig. 2. Photograph of the driving mechanism for the adaptor.

the whole Soller slit assembly until the X-ray beam falling on the crystal produces a shadow on a fluorescent screen. A backstop is mounted on a sleeve fitting over the collimator shaft, and permits the counter to record spectra with angles as low as  $3^{\circ} 20$ .

A photographic view of the drive portion of this adaptor is given in Fig. 2. The intensity of the reflection is obtained by continuously counting through the reflecting position, and subtracting out the corresponding background. If the intensity thus obtained is of insufficient accuracy due to too few counts being recorded, a slower rocking velocity may be used. The resolution obtained may be improved by inserting a slit in front of the Geiger counter window, but the intensity loss which ensues often appears to make attempts at greater resolution incompatible with high accuracy in the intensity measurement. Upper layers could be measured by remounting the Geiger tube so as to be able to slide it along a lightweight arc attached to the present support. This arc would be part of a circle with the crystal as center. However, no attempt has yet been made to do this.

This adaptor has now been in use in this laboratory for over one year.

We wish to thank P. Kelleher for his careful construction of this device.